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ORIGINAL ARTICLE

Effect of Sintering Temperature on the Physical Properties of $Ba_{0.6}Sr_{0.4}TiO_3$ Prepared by Solid-State Reaction

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ABSTRACT – Barium Strontium Titanate (BST) ceramic materials are widely used in electronic devices due to their stable operation at high temperatures, high tunability, low tangent loss, low DC leakage, and alterable curie temperatures. While pure BST materials are usually produced at high sintering temperatures (1250 °C), there are limited studies on the temperature and duration of the sintering process to produce pure BST, synthesised from micro or even nano-sized raw materials. This study aims to determine the effective sintering temperature for producing pure BST material using a mixture of raw materials with a mean particle size of 0.4 µm after milled for 58 hours. The $BaCO_3$, $SrCO_3$, and TiO_2 materials as raw materials for $Ba_{0.6}Sr_{0.4}TiO_3$ synthesis were milled for 58 hours to produce a homogeneous mixture with a mean particle size of 0.4 µm. Sintering was carried out in a temperature range of 500-1100 °C for 1 hour. This study investigates the impact of sintering temperature on the physical properties and the purity of $Ba_{0.6}Sr_{0.4}TiO_3$ powder using the x-ray diffraction method. The results showed that the $Ba_{0.6}Sr_{0.4}TiO_3$ phase was formed at a sintering temperature of 700 °C. Pure BST material was formed at the sintering temperature of 1000 °C with a crystallite size of 41 nm. Whereas at a higher sintering temperature (1100 °C), the pure BST material formed produced a larger crystallite, sized at 43 nm with cubic structure. The synthesis temperature and duration recorded in this research are lower than recorded in the BST material preparation using the solid-state method. The results of this study indicate that the sintering temperature greatly affects the purity, crystal system and crystallite size of the $Ba_{0.6}Sr_{0.4}TiO_3$ material produced. The sintering temperature of 1100 °C produces $Ba_{0.6}Sr_{0.4}TiO_3$ material with the best physical properties because it has a cubic-shaped crystal system and the largest crystal size.

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INTRODUCTION

Barium Strontium Titanate (BST), $Ba_{(x)}Sr_{(1-x)}TiO_3$, $0<x<1$, is a ferroelectric material with a perovskite structure. This material composes of barium titanate ($BaTiO_3$) and strontium titanate ($SrTiO_3$). When $SrTiO_3$ is added to $BaTiO_3$, Sr ion replaces Ba ion to form the BST material structure. Barium strontium titanate ceramic material is widely used in electronic devices [1]. As bulk ceramics and thin films, this material has a unique combination of large dielectric constant. It demonstrates a stable operation at high temperature, high tunability, low loss tangent, low DC leakage, and alterable curie temperature [2]. The uniqueness of the BST material is attributed to several factors, such as the Ba / Sr ratio, the synthesis method, and the particle and crystal size [3]. $Ba_{(x)}Sr_{(1-x)}TiO_3$ ceramics with various compositions of x (Ba / Sr ratio) has been widely studied; for example x = 0.2, 0.3, 0.4, 0.5, and 0.6 [4]. X-ray diffraction (XRD) results showed BST crystals formation in all variations of x; after the sintering process was carried out at 1000 °C for 3 hours. However, there was still a secondary phase (Sr_2TiO_4, $SrTiO_{10}$, and $Sr_3Ti_2O_7$). This can be improved by increasing the sintering temperature. The secondary phase can be removed by increasing the sintering temperature. Increasing the concentration of x causes a transformation of the crystal system from tetragonal to cubic. The grain size and density of the sample decrease with increasing concentration x because Sr is having a smaller radius than Ba. Curie temperatures for samples with x = 0.2, 0.3, 0.4, 0.5, and 0.6 were 70 °C, 28.7 °C, -8.4 °C, -45.5 °C, and -82.6 °C, respectively. Curie temperature decreased with increasing x concentration. The highest dielectric constant found in sample with x = 0.3 [4]. Furthermore, the high dielectric and pyroelectric properties of the composition $Ba_{(x)}Sr_{(1-x)}TiO_3$ with $0.3 \leq x \leq 0.5$ resulted in the Curie temperature (TC) or ferroelectric-paraelectric phase transformation temperature decrease, near to room temperature (25 °C) [5].

$Ba_{(x)}Sr_{(1-x)}TiO_3$ materials are generally synthesised by hydrothermal method, sol-gel method, and conventional solid-state reaction method [6]. The BST material synthesised by the hydrothermal and sol-gel method produces crystals less than 100 nm. Subsequently, it produces residual hydroxide ions, which resulted in the formation of intergranular pores [7]. The mechanochemical or solid-state method is the most used method for BST nanoparticles large-scale production [8]. The solid-state method has several advantages; it uses low-cost raw materials, simple synthesis processes, and the ability to produce fine particles [8]. During solid-state processes such as high-energy ball milling, the steps that occur during solid-state processes, specifically welding, deformation, and fracture of powder raw materials, are repeated [9].

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The mechanical activation during solid-state reaction with mechanical milling increases the raw material specific surface area due to the destruction of agglomerates and particles of the initial precursors [10]. In this regard, the mechanical milling process can produce particles sized > 1 μm. In general, the microstructure of the particle and crystallite size of mechanical milling products were influenced by the characteristics of the raw materials used, duration and heating temperature during the mechanical milling process [11].

In recent years, several studies have focused on the effect of sintering temperature on the synthesis of Barium Strontium Titanate (BST). In general, the increase of the temperature and duration time of the sintering process affected the BST material's purity and increased the crystallite size [12]. Pure BST materials are usually produced at high sintering temperatures [4], [12]–[18]. Sandi et al. carried out a sintering process at 1200 °C for 2 hours to produce pure BST material by the solid-state reaction method [8]. Yustanti et al. reported that the sintering raw materials measuring an average size of 2.4 μm at a temperature of 1200 °C produced pure BST material without the presence of impurities [14]. In the meantime, barium strontium titanate ceramic material was synthesised from fine constituent powders produced from high energy ball milling processes at sintering temperatures 1200-1350 °C, as reported by Mudinepalli et al. [12]. Budkhod et al. performed sintering at temperatures between 1050-1350 °C to produce $Ba_{0.7}Sr_{0.3}TiO_3$ ceramics using a hybrid method between solid-state reaction and sol-gel combustion methods using urea as a fuel [15]. Meanwhile, Gate et al. conducted a sintering process at a temperature of 1100°C for 3 hours to produce a pure $Ba_{0.6}Sr_{0.4}TiO_3$ material using the sol-gel method [17]. Lastly, Zhu et al. synthesised barium strontium titanate glass and ceramics using the sol-gel method and was sintered at temperatures between 1000 and 1150°C [18].

The high sintering temperature to produce pure BST material has prompted various studies to produce pure BST materials at lower sintering temperatures using glass, polymer and inorganic additives [19]. One way to produce BST material at low sintering temperatures is to add Li_2O material, as reported by Zhang et al.[20]. Zhang et al. investigated the effect of adding Li_2O on the sintering temperature of commercial BST. It was found that by adding 0.5 wt%, Li_2O could reduce the sintering temperature to 900 °C without decreasing the ceramics performance. The sintering temperature of BST materials was reduced from 1350 °C to 900 °C by adding Li [21]. However, the XRD test showed two secondary phases as impurities caused by the addition of lithium. Besides, this research method has many limitations when applied to commercial production because it requires a very high cost [19], [21].

However, the BST material sintering ability decreased along with the broader particle size distribution [22]. Accordingly, reducing the particle size is another way that can be used to speed up or shorten the sintering time. The sintering temperature is reduced with smaller raw material particles. The smaller the raw material particle size, the faster the grain becomes coarse due to the high particle surface energy compared to the large grain size distribution in the batch under the same sintering conditions [23]. In another reference, the raw material with smaller dimensions causes a larger contact surface so that the sinterability increases due to the maximal diffusion process [24]. Therefore, this study uses a vibratory ball milling for 58 hours to mix $BaCO_3$, $SrCO_3$, and TiO_2 to obtain a homogeneous mixture with a mean particle size of 0.4 μm. This research aims to determine the effective sintering temperature to produce pure BST material using a mixture of $BaCO_3$, $SrCO_3$, and TiO_2 ball-milled for 58 hours.

METHODS AND MATERIALS

$Ba_{0.6}Sr_{0.4}TiO_3$ was synthesised from $BaCO_3$, $SrCO_3$, and TiO_2 powders using the mechanical alloying method with a purity level of 99%. The powers were obtained from Sigma-Aldrich. The particle size analyser (PSA) test results in Table 1 show the $BaCO_3$, $SrCO_3$, and TiO_2 powders average sizes. Meanwhile, the results of XRD tests on TiO_2, $SrCO_3$, and $BaCO_3$ powders are shown in Figure 1(a). The diffraction patterns of TiO_2, $SrCO_3$ and $BaCO_3$ powders are in line with the diffraction patterns of TiO_2, $SrCO_3$ and $BaCO_3$ in the Inorganic Crystal Structure Database (ICSD), respectively, with numbers 98-009- 6946, 98-016-6088, and 98-016-6090. The Rietveld analysis using High Score Plus software shows that the TiO_2 powder has tetragonal-shaped crystal systems while the $SrCO_3$ and $BaCO_3$ powders have orthorhombic crystal systems.

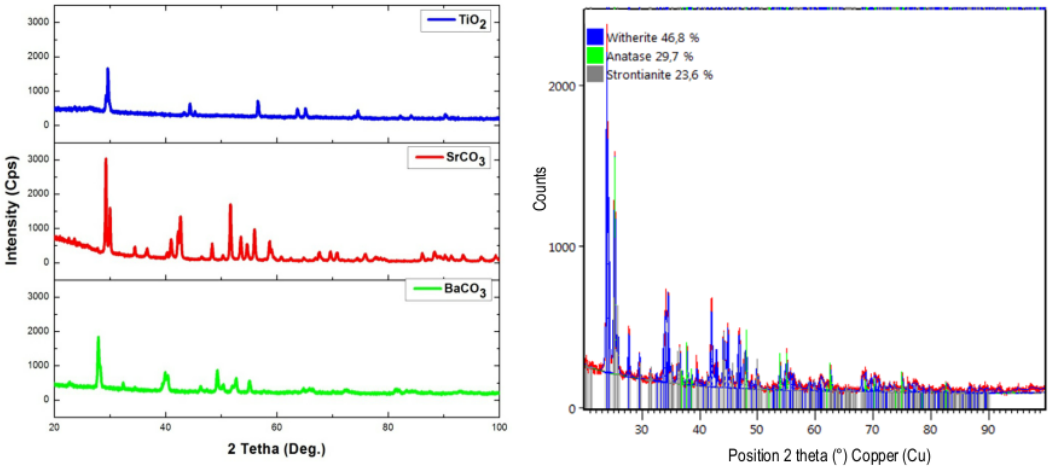
Table 1. Average particle size of $BaCO_3$, $SrCO_3$ and TiO_2.

Raw materials	Average particle size (μm)
$BaCO_3$	1.979
$SrCO_3$	3.182
TiO_2	0.795

In this research, $BaCO_3$, $SrCO_3$ and TiO_2 powders were subjected to a wet milling process using a vibratory ball milling machine with a composition of 41.63 grams each, 20.76 grams, and 28.08 grams for 58 hours. The ball to powder ratio (BPR) in the milling process is 10:1. After 58 hours, the mixed $BaCO_3$, $SrCO_3$ and TiO_2 were tested for PSA and XRD. The PSA test results showed that the powder mixture of $BaCO_3$, $SrCO_3$ and TiO_2 had a particle size of 0.4 μm. Meanwhile, according to the Rietveld analysis using the High Score Plus software, the XRD test results are shown in Figure 1(b). Figure 1(b) shows no change in the diffraction pattern of the powder of TiO_2, $SrCO_3$ and $BaCO_3$. The crystal sizes of $BaCO_3$, $SrCO_3$ and TiO_2 calculated using The Williamson-Hall method are 48 nm, 61 nm, and 71 nm, respectively.

After characterising the mixture of milled TiO_2, $SrCO_3$, and $BaCO_3$ powders, the next step is sintering the powder mixture of TiO_2, $SrCO_3$ and $BaCO_3$. The sintering process was carried out in the electric chamber furnace (Nabertherm

N31/H) with temperature variations of 500 °C, 600 °C, 700 °C, 800 °C, 900 °C, 1000 °C, and 1100 °C in the air under atmospheric pressure up to 1 hour. After the sintering process, characterisation was carried out using the XRD methods. The Philips XRD was also used to analyse the resulting phase and crystallite size. The step-scan method was performed to record the x-ray diffraction patterns. The intensity data during the scanning was taken every 2 seconds for each step on the diffraction angle of 0.005°. The Rietveld analysis was conducted using the High Score Plus software. The description of the diffraction line profiles at Rietveld refinement was achieved using the pseudo-Voigt function.



(a) (b)

Figure 1. X-ray diffraction patterns of TiO_2, $SrCO_3$ and $BaCO_3$ powders (a) before and (b) after 58 hours of milling.

RESULTS AND DISCUSSION

Figure 2 shows the diffraction pattern of $BaCO_3$, $SrCO_3$, and TiO_2 powder mixture at each sintering temperature. From the figure, the $Ba_{0.6}Sr_{0.4}TiO_3$ phase was not formed in the material sintered at temperatures of 500 °C and 600 °C. Meanwhile, while the $Ba_{0.6}Sr_{0.4}TiO_3$ phase was formed at temperatures of 700 °C, 800 °C and 900 °C, other compound phases, $BaCO_3$, $SrCO_3$, TiO_2 and $BaTiO_3$, were observed. Moreover, a single-phase $Ba_{0.6}Sr_{0.4}TiO_3$ was formed at temperatures of 1000°C and 1100°C.

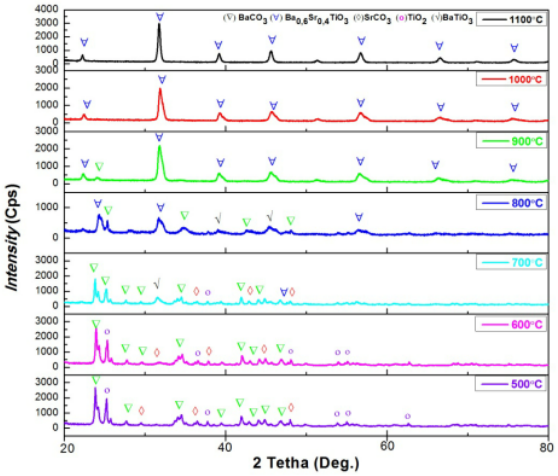


Figure 2. X-ray diffraction patterns of the sintered samples.

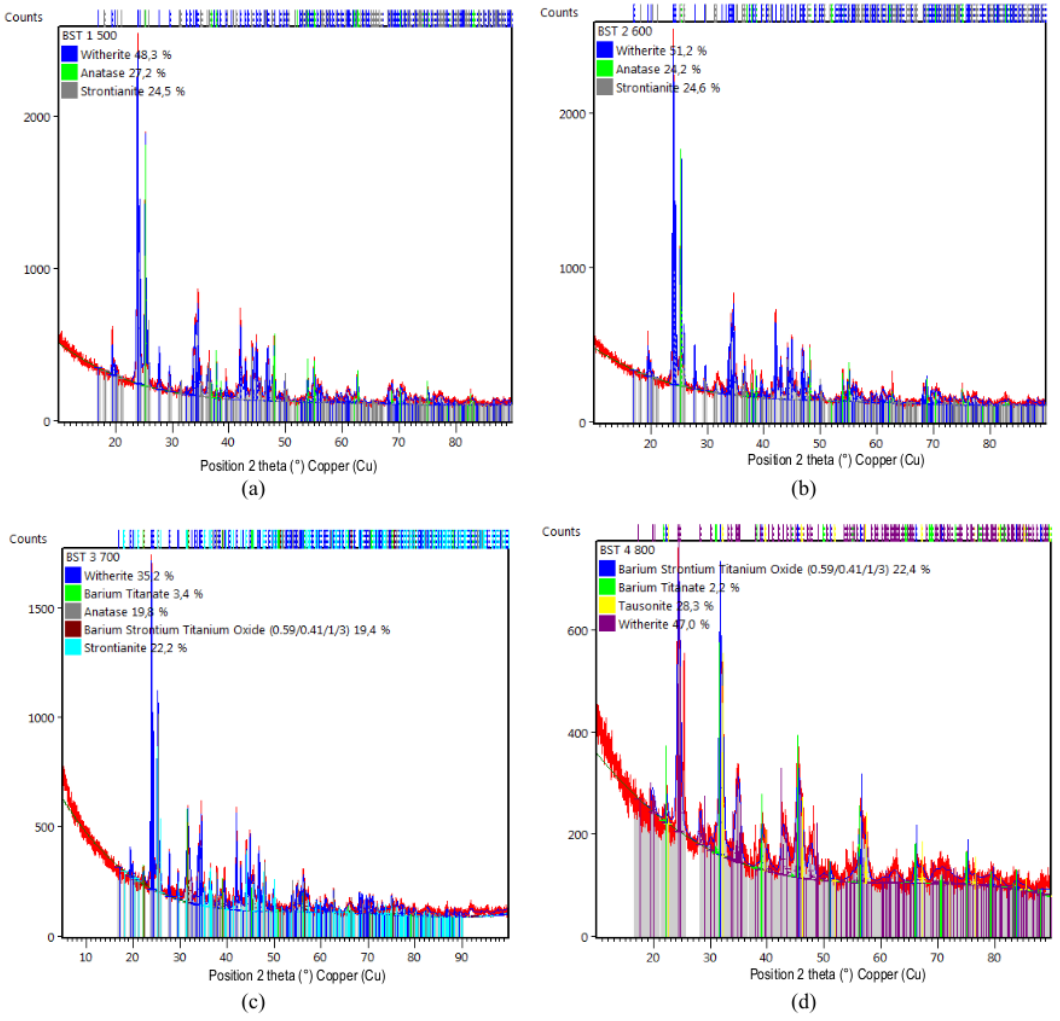
Figure 3 shows the Rietveld analysis results using the High Score Plus software based on X-ray diffraction obtained from the test results. Figure 3(a) shows the x-ray diffraction pattern on a powder that has been sintered at 500 °C. The x-ray diffraction pattern shows that the $Ba_{0.6}Sr_{0.4}TiO_3$ phase was not formed. In this light, the phases formed at 500 °C sintering temperature variations are $BaCO_3$ (witherite), TiO_2 (anatase), and $SrCO_3$ (strontianite), with the respective percentage of 48.3%; 27.2%, and 24.5%. Meanwhile, the crystallite size formed in $BaCO_3$, TiO_2, and $SrCO_3$ was 54.7 nm, 125.6 nm, and 80.3 nm. The x-ray diffraction pattern of the sintered powder at 600 °C is shown in Figure 3(b). At

600 °C, $Ba_{0.6}Sr_{0.4}TiO_3$ was not formed and still in the $BaCO_3$, TiO_2, and $SrCO_3$ phases, with 51.2%; 24.2% and 24.6%, respectively. The sizes of the crystallites formed after sintering $BaCO_3$, TiO_2, and $SrCO_3$ at 600°C for are 56.4 nm, 104.3 nm, and 122.3 nm, respectively.

Figure 3(c) shows the x-ray diffraction pattern of a mixture of $BaCO_3$, $SrCO_3$, and TiO_2 powders sintered at 700 °C. At a temperature of 700 °C, the $Ba_{0.6}Sr_{0.4}TiO_3$ phase was formed with a percentage of 19.4% and a crystallite size of 15.3 nm. However, at 700 °C, the $BaCO_3$, $SrCO_3$ and TiO_2 phases were still found at a respective percentage of 35.2%, 22.2%, and 19.8%, and crystallite sizes of 58.1 nm; 65.6 nm and 66.2 nm, respectively. Apart from $Ba_{0.6}Sr_{0.4}TiO_3$, $BaCO_3$, $SrCO_3$ and TiO_2 phases, a new phase, $BaTiO_3$ at 3.4% and crystallite size of 38 nm, was also formed.

Figure 3(d) shows that the crystallite percentage increased to 22.4%, and its size increased to 30.3 nm at the $Ba_{0.6}Sr_{0.4}TiO_3$ phase when sintering at 800 °C. In this variation, the crystal size of $Ba_{0.6}Sr_{0.4}TiO_3$ is two times the crystallite size formed at 700 °C sintering temperature. Meanwhile, the $BaTiO_3$ phase percentage decreased to 2.2%, accompanied by a decrease in crystal size to 19.5 nm. $BaCO_3$ phases were still found with a composition of 47% with a crystallite size of 40 nm. Apart from $Ba_{0.6}Sr_{0.4}TiO_3$, $BaCO_3$, and $BaTiO_3$ phases, a new phase, $SrTiO_3$ was also formed at 28.3% and crystallite size of 32.6 nm.

Figure 3(e) shows the x-ray diffraction pattern at a product sintering temperature of 900 °C. The $Ba_{0.6}Sr_{0.4}TiO_3$ phase formed in this variation experienced a significant increase with a percentage of 94.6% and a crystallite size of 40.1 nm. Simultaneously, the other 5.4% is the $BaCO_3$ phase with a crystallite size of 21.5 nm. The single phase of $Ba_{0.6}Sr_{0.4}TiO_3$ was formed perfectly at various sintering temperatures of 1000 °C and 1100 °C, as shown in Figure 3(f) and 3(g). $Ba_{0.6}Sr_{0.4}TiO_3$ with crystallites size of 30 nm and 43 nm resulted in sintering temperatures of 1000 °C and 1100 °C.



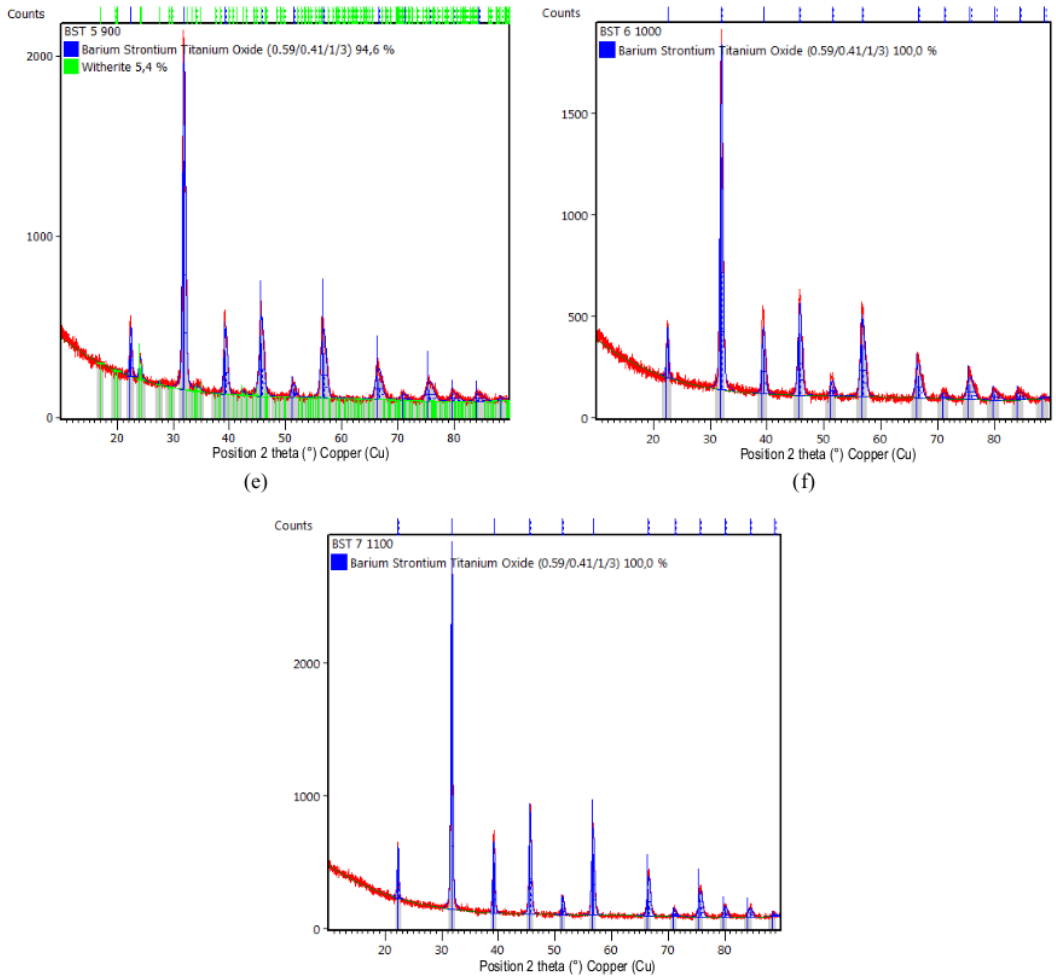


Figure 3. X-ray diffraction patterns of sintered samples at (a) 500 °C, (b) 600 °C, (c) 700 °C (d) 800 °C, (e) 900 °C, (f) 1000 °C and (g) 1100 °C.

The results of this study indicate that the $Ba_{0.6}Sr_{0.4}TiO_3$ material has begun to form at a sintering temperature of 700 °C. However, a secondary phase is still formed and completely into single-phase $Ba_{0.6}Sr_{0.4}TiO_3$ at 1000°C and 1100°C. The effect of sintering temperature on the phase composition (%) and crystallite sizes of $Ba_{0.6}Sr_{0.4}TiO_3$ formed is shown in Figure 4. The impurity phase disappears proportionately with the increase in sintering temperature. The kinetic energy in atoms increases due to higher sintering temperatures. The higher temperature makes it easier for the atoms to interact and bond with each other, causing the impurity phase to disappear [12], [17]. Besides, increasing the sintering temperature causes the atomic bonds to become stronger. Thus, sintering carried out at 1000°C and 1100°C resulted in forming a single phase of $Ba_{0.6}Sr_{0.4}TiO_3$ which is more stable. Increasing the sintering temperature will result in a recrystallisation process in the raw materials ($BaCO_3$, $SrCO_3$, and TiO_2) so that a more stable $Ba_{0.6}Sr_{0.4}TiO_3$ phase is formed. An increase in sintering temperature will produce $Ba_{0.6}Sr_{0.4}TiO_3$ with a higher phase composition (%). The crystallite sizes also increased, as reported in previous studies [12], [17], [25], [26].

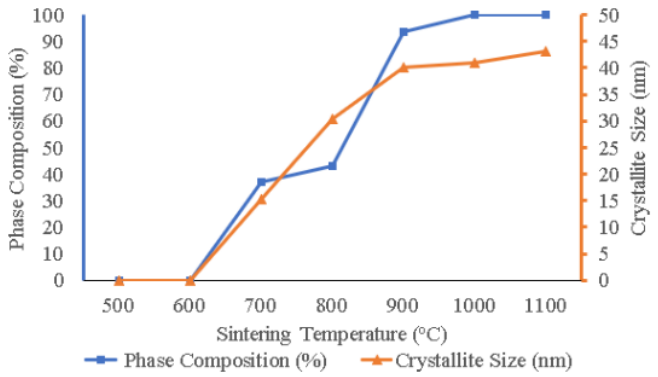


Figure 4. The effect of sintering temperature on the physical properties of $Ba_{0.6}Sr_{0.4}TiO_3$.

The use of $BaCO_3$, $SrCO_3$, and TiO_2 powder mixture, with a particle size of 0.4 µm, has been proven to produce $Ba_{0.6}Sr_{0.4}TiO_3$ material at lower sintering temperatures with a faster duration (1 hour). This is supported by [27] and [24] study, which revealed that reducing the particle size of raw materials is another method that can be used to speed up sintering and shorten sintering time. The sintering temperature is reduced with smaller raw material particles. The smaller the raw material particle size, the faster the grain becomes coarse due to the high particle surface energy compared to the large grain size distribution in the batch under the same sintering conditions [27]. Previous research [24] mentioned raw materials with smaller dimensions causes a larger contact surface so that the sinterability increases due to the maximum diffusion process. In this study, sintering carried out at temperatures of 1000 °C and 1100 °C for 1 hour produced single-phase $Ba_{0.6}Sr_{0.4}TiO_3$. In this study, single-phase $Ba_{0.6}Sr_{0.4}TiO_3$ resulted in lower sintering temperature and shorter sintering duration compared to the results of the study conducted by [4], [12], [14], [17], [26]. These studies' results showed the single-phase formation of $Ba_{0.6}Sr_{0.4}TiO_3$ was carried out in a sintering temperature range of 1100 °C-1350 °C with a sintering duration of ≥ 2 hours.

In addition, [4], [12], [14], [26], [28] have clearly shown that the decrease in calcination or sintering temperature is due to the reduction in the small particle size of the raw materials; ranging from submicrometer or even to the nanoscale. The use of a smaller particle size increases the contact area and decreases the reactants contact distance, thereby increasing the overall reaction kinetics. The results of this study indicate that the sintering temperature greatly affects the purity, crystal system, and crystallite size of the material $Ba_{0.6}Sr_{0.4}TiO_3$ produced. The effect of sintering temperature on the physical properties of $Ba_{0.6}Sr_{0.4}TiO_3$ formed is shown in Table 2. The higher the sintering temperature resulted in a sharper peak intensity at $Ba_{0.6}Sr_{0.4}TiO_3$, which indicates an increase in crystal size. Accordingly, the higher the sintering temperature used, the higher the crystal size of the material $Ba_{0.6}Sr_{0.4}TiO_3$ formed. This is supported by the studies of [4], [17], [26], [29]. Thus, increasing the sintering temperature increased the atoms kinetic energy to diffuse and make the atoms react and bond perfectly. This finding is supported by other studies [29]–[32] that found that the crystallites produced have a larger size and higher crystallinity.

Table 2. The Physical Properties in The Formation of $(Ba_{0.6}Sr_{0.4})TiO_3$.

Sintering temperature (°C)	Phase	Composition phase (%)	Crystal system	Crystallite size (nm)
500	$BaCO_3$	48	Orthorhombic	54.7
	TiO_2	27	Tertragonal	125.6
	$SrCO_3$	25	Orthorhombic	80.3
600	$BaCO_3$	51.2	Orthorhombic	56.4
	TiO_2	24.2	Tertragonal	104.3
	$SrCO_3$	24.6	Orthorhombic	122.3
700	$BaCO_3$	35.2	Orthorhombic	58.1
	TiO_2	19.8	Tertragonal	19.8
	$SrCO_3$	22.2	Orthorhombic	65.6
	$BaTiO_3$	3.4	Orthorhombic	38
	$(Ba_{0.67}Sr_{0.33})$ TiO_3	19.4	Tertragonal	15.3
800	$BaCO_3$	47	Orthorhombic	40
	$BaTiO_3$	2.2	Orthorhombic	19.5
	$SrTiO_3$	28.3	Orthorhombic	32.6
	$(Ba_{0.67}Sr_{0.33})$ TiO_3	22.4	Tertragonal	30.3
900	$BaCO_3$	5.4	Orthorhombic	21.5
	$(Ba_{0.67}Sr_{0.33})$ TiO_3	94.6	Tertragonal	40.1
1000	$(Ba_{0.59}Sr_{0.41})$ TiO_3	100	Cubic	41
1100	$(Ba_{0.6}Sr_{0.4})$ TiO_3	100	Cubic	43

In addition, an increase in the sintering temperature in this study caused changes in the crystal system of the $Ba_{0.6}Sr_{0.4}TiO_3$ material. At temperatures of 700 °C, 800 °C, and 900 °C, the crystal system in the material $Ba_{0.6}Sr_{0.4}TiO_3$ is tetragonal. Whereas at temperatures of 1000 °C and 1100 °C, the crystal system in the $Ba_{0.6}Sr_{0.4}TiO_3$ is cubic with a higher peak intensity with increasing sintering temperature. The $Ba_{0.6}Sr_{0.4}TiO_3$ with cubic structure increased the dielectric properties of the material $Ba_{0.6}Sr_{0.4}TiO_3$ produced [12], [17], [33]. Besides the crystal structure, the dielectric properties of the $Ba_{0.6}Sr_{0.4}TiO_3$ material are also influenced by the crystal size, where the dielectric property increases with the crystallite size [12], [29], [34]. In this study, the sintering temperature of 1100 °C produces $Ba_{0.6}Sr_{0.4}TiO_3$ material with the best physical properties because it has a cubic-shaped crystal system and the largest crystal size.

CONCLUSION

The effect of sintering temperature on the physical properties of $Ba_{0.6}Sr_{0.4}TiO_3$ material synthesised with 0.4 µm raw materials (and milled up to 58 hour) using the solid-state reaction method has been investigated. The use of raw materials, a mixture of $BaCO_3$, $SrCO_3$, and TiO_2 with a particle size of 0.4 µm could produce $Ba_{0.6}Sr_{0.4}TiO_3$ material at a lower sintering temperature of 700 °C. Moreover, the single-phase $Ba_{0.6}Sr_{0.4}TiO_3$ can be produced at temperatures of 1000 °C and 1100 °C. Increasing the sintering temperature in this study resulted in $Ba_{0.6}Sr_{0.4}TiO_3$ material with higher purity as marked by the disappearance of the $BaTiO_3$, $BaCO_3$, $SrCO_3$, and TiO_2 phases. Higher sintering temperature increased the crystal size and changed the crystal system from tetragonal to cubic. The sintering temperature of 1100 °C produces $Ba_{0.6}Sr_{0.4}TiO_3$ material with the best physical properties because it has a cubic-shaped crystal system and the largest crystal size.

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